Manufacturing Methods and Technology Engineering

for

"Growth of Large Diameter Nd:YAG
Laser Crystals"

by

R. Uhrin and R.F. Belt

Semiannual Progress Report
October 1, 1979 to April 1, 1980
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Litton Industries, Inc.
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This report describes initial experiments on the growth of two inch diameter Nd:YAG laser crystal boules by the Czochralski method. Power was supplied through a 450 kHz, 120 kVA, RF generator to heat a 4.5 x 4.5 inch cylindrical iridium crucible. Melt charges of 4.3 kg were employed which contained a 1.1 atomic percent of Nd. The first few runs presented difficulties in enlarging the seed crystal to the finished diameter. Most boules

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were strained highly and cracked spontaneously. A technique of gradual enlargement of the seed worked more satisfactorily and allowed short sections of two inch boules to be grown. Cracking of boules is related to blossom formation after seeding and is connected with large radial gradients in the melt. Measurements were made to define and minimize these gradient for increased chances of large boule growth. Seven growth runs were completed this period but no suitable material for rods was obtained.

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The objective of this program is to investigate methods for increasing the boule diameter of Nd:YAG production growth runs without deterioration of laser rod quality. A goal of two inches would nearly double present rod yields.

PURPOSE

The growth of Nd:YAG for laser rods is performed exclusively by the Czochralski method. Production boules are currently 1.25-1.50 inches in diameter. The purpose of this program is to obtain a larger yield of high quality rods by increasing the diameter of the grown boule. Preliminary investigation has shown that a suitable goal of 2.0 inches could nearly double rod yield.

This program consists of several parts including crystal growth, rod fabrication, and passive testing for quality. Laser rods provided under the program are required to meet existing military specifications.

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FOREWORD

This final technical report on Manufacturing Methods and Technology Engineering for "Growth of Large Diameter Nd:YAG Laser Rods" was prepared by the Airtron Division of Litton Industries, Inc., Morris Plains, New Jersey 07950 under Contract No. DAAB07-77-C-0375 for the Solid State and Injection Laser Team of the U.S. Army Electronics Research and Development Command, Night Vision and Electro Optics Laboratory, Fort Belvoir, Virginia 22060.

The program was initiated by Mr. John Strozyk and monitored by Mr. William Comeyne. The growth of Nd:YAG for laser rods is performed exclusively by the Czochralski method. Production boules are currently 1.25-1.50 inch in diameter. The purpose of this program is to obtain a larger yield of high quality rods by increasing the diameter of the grown boule. Preliminary investigation has shown that a suitable goal of 2.0 inches could nearly double rod yield for small sizes.

This project consists of several parts including crystal growth, rod fabrication, and passive testing for quality. Laser rods provided under the program are required to meet existing military specifications for a (4.3 x 43) mm rod used in the GVS-5 and other Army programs.

1.0 Introduction

Large single crystals of neodymium doped yttrium aluminum garnet (Nd:YAG) are required for many military and civilian applications of optically pumped solid state lasers. From its discovery in 1964 until the present time, the most expedient method of obtaining such crystals of laser quality has been by means of the Czochralski growth procedure. As currently practiced this method consists of seeding and pulling a crystal from a melt contained in an iridium crucible. The crucible is heated by means of R.F. induced currents. While the process is a good one it has remained virtually unchanged except for improvements in diameter and control systems. Early work on Nd:YAG growth has been described in several publications.[1-3] The Defense Department through the U.S. Army has sponsored two production engineering programs connected with the timely growth[4] and laser rod fabrication[5] of Nd:YAG. These programs have determined essentially the crystal size, boule yield, fabrication methods, and hence cost of laser rods. In general terms because boule growth and yields are limited, the larger the rod size the higher the cost. Unfortunately this relationship is not linear and laser rods of a size greater than (7 x 75)mm still merit a premium price.

At the conclusion of the growth program[4] on Nd:YAG, a production process was developed which yielded boules of 1.25-1.50 inches in diameter. For nearly ten years this process has

remained the same. In the past few years an increased demand for laser rods has engendered an examination of procedures to increase yields. A concurrent objective is the lowering or stabilization of growth costs. The principal contributions toward the cost of a laser rod are iridium, electrical power, materials, and labor. Thus any process which limits or eliminates any of these would be beneficial. During the natural evolution of any growth technology, the trend has been to grow larger crystals. A valid question has been asked often; why not grow larger boules of Nd:YAG?

The justification for larger boules of Nd:YAG is based on the need for greater yields of high quality material at lower costs. This can be accomplished by the following methods:

1. Grow longer crystals at current production diameters and quality.
2. Grow larger diameter crystals at the same length and quality.
3. Improve the optical quality of boules by maintaining the melt composition fixed. This may combine any advances made in 1 and/or 2.

If objective 1 is accomplished the growth rate still remains fixed and not much is gained. This is further complicated by the Nd level in the crystal which constantly increases and eventually causes high strain or exceeds the concentration specification. If objective 3 is accomplished a substantial

improvement results, but the time frame for realizing such a condition is certainly several years. Thus the most promising alternative is objective 2; to grow larger diameter crystals. An increase in diameter to about 50 mm would almost double the rod yield from a boule and seems to be within capabilities based on recent experiments.

Early experiments were begun at Airtron nearly two years ago with moderate success. These results led to an initiation of the present program to refine the technique for production purposes. The increase of boule diameter of any Czochralski grown crystal is a formidable task. Good methods have been developed for silicon, GGG, and sapphire over a period of years. The degree of difficulty is associated closely with the operating temperatures, number of chemical components, and factors which govern melt behavior. Nd:YAG growth is complicated by a melting point of 1975°C, a three component system, low distribution coefficient of Nd, faceting phenomena, and high melt thermal convection. In addition the growth rate of Nd:YAG from the melt is a rather low 0.5 mm/hr. This places an xtremely high demand on the temperature control system. Fluctuations of many °C cannot be tolerated during the entire growth cycle of 2-3 weeks. At the present time there is no known method to increase growth rate without a sacrifice in quality. Hence for any planned increase of boule diameter, all the usual problems

are not only present but also aggravated. In spite of inherent difficulties with Nd:YAG, it is safer to follow the Czochralski growth route than an untried path of any entirely different procedure.

2.0 Experimental

Early experiments on large diameter growth of Nd:YAG began nearly two years ago. At that time the largest crucibles of iridium were 3.5 x 3.5 inch. These crucibles could be charged and heated with a 40 kW, 92 kVA, 450 kHz RF generator. In preparation for the current program, the scaled increase of diameter to about 2 inches dictated that the crucible size should be approximately 4.0-4.5 inches in diameter. For this purpose an RF generator of larger capacity was employed. All of the runs were made with 50 kW, 120 kVA, 450 kHz RF generators. Since all of our production capability consists of the lower power capability equipment, early experiments were conducted with such equipment. Results of these growth runs indicated that the production equipment would lack the capacity to perform adequately near its operating limit. Thus additional equipment for experimental as well as production requirements is expected to have the higher power capability.

2.1 Growth Station Design

Figure 1 represents the typical growth station used for growth of the large diameter crystals. Power is supplied by a 450 kHz RF generator capable of supplying a

Figure 1   Growth Station for Czochralski Nd:YAG Crystals

maximum of 50 kW directly to the iridium growth crucible which acts as the susceptor. The growth furnace is surrounded by a water-cooled enclosure connected to a water-cooled support table. The water-cooled system is required due to the inordinate amount of heat radiated from the growth furnace. The growth furnace arrangement consists of an iridium crucible centered in the RF induction coil. The crucible is surrounded by granular zirconia insulation and supported by zirconia tubing. The area above the crucible is enclosed by an alumina tube having an alumina cover through which the seed rod passes. The crucible is also covered with an iridium ring which reduces the heat loss from the melt surface during growth and adjusts the radial melt temperature gradient.

2.2 Crucibles

In order to provide a situation for experimental growth similar to that existing in the production growth of smaller diameter crystals, the crucible size has been optimized at 4.5 inch diameter and 4.5 inch high. This insures that for a given length of crystal the neodymium dopant concentration typifies that of production crystals and permits the duplication of crystal growth rate. The charge for a crucible of this size capacity is approximately 4300 grams. The expected weight of the pulled crystal is about 1 kg so no more than 20-25% of the melt is removed. Figure 2 illus-

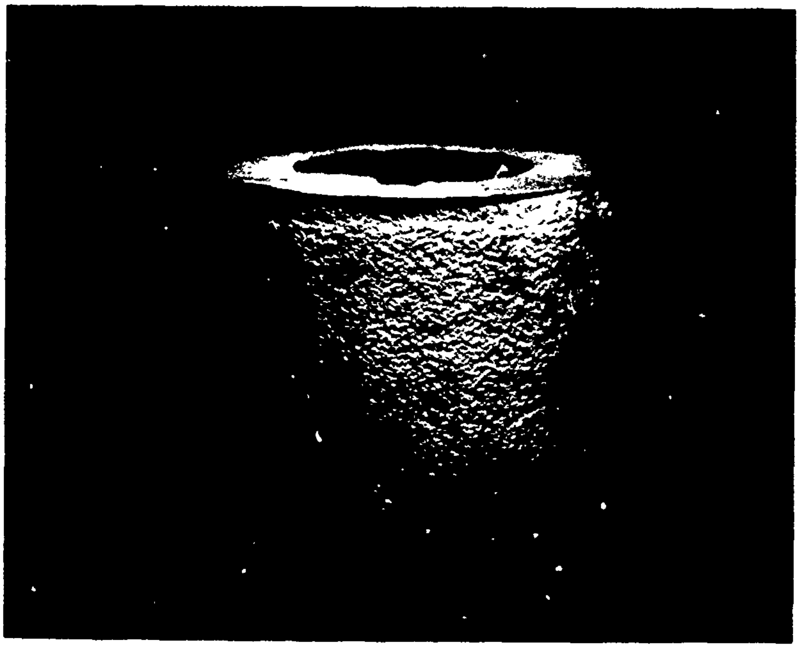

Figure 2 Iridium Crucible and Cover,
4.5 x 4.5 Inches

trates the appearance of the crucible and its cover.

2.3 Raw Materials

Oxides used for experimental work are obtained from supplies used in the production growth area. These are readily available from commercial vendors at grades of 5-9 and 6-9 purity. In the case of the yttrium and neodymium oxides the purity refers only to the rare earth oxide assay, however. Thus care must be exercised to insure that contaminants do not affect crystal growth or laser performance of fabricated rods.

2.4 Procedures

First the growth furnace is constructed by carefully aligning ceramic elements and the crucible for cylindrical symmetry. The oxides are blended to a homogeneous mixture and are then added to the crucible. In order to initiate growth, the melt temperature is adjusted to maintain the seed diameter when contact is made with the melt surface. If a diameter increase or decrease occurs, the temperature is adjusted upward or downward as required to maintain seed diameter. When pulling commences, the automatic diameter and temperature control is initiated and growth continues until the desired crystal length is obtained. The growth is then terminated and the entire furnace is cooled to room temperature over a period of several days.

Following completion of the growth run the crucible

is thoroughly cleaned. This is accomplished by coring out the solidified melt with a diamond impregnated core drill. The crucible is then submerged in a container of molten lead fluoride which has an appreciable solubility for YAG. This treatment normally cleans the crucible to the point where a small amount of cleaning in acids readies it for use in the next growth cycle. Prior to this, however, it is tested for leaks and is repaired if necessary to prevent failure in the subsequent growth run.

2.5 Gradient Control

The most critical problem to be overcome in the growth of large diameter crystals is control of temperature gradients in the growth furnace and melt. This is accomplished by proper positioning of ceramic insulating components around the crucible and in the area above the crucible into which the crystal is pulled during growth. It appears at this time that the most desirable condition is to have a radial melt temperature gradient of 25-30°C per cm of radius. This is established reproducibly by the positioning of the crucible in the induction coil. It has not been completely defined because some changes of shape occur in the crucible. Characterization of this gradient is performed with an optical pyrometer for a given station design and growth results dictate the acceptability of each growth station design. Characterization of the vertical gradient in the

afterheater chamber is somewhat more difficult and has not been attempted. However a design similar to a production station is utilized. The main objective here is to prevent cracking during cooling of the crystal.

The constraints put on growth by the larger crucible size are significant since a large change in the radial melt temperature gradient results. A more flat solid/liquid growth interface shape is difficult to achieve and this leads to defects (referred to as "blossoms") fanning out from the central core. This causes degradation in crystal quality and premature cracking as the crystal grows out to final diameter.

3.0 Growth Run Results

Since the growth program was initiated continual progress has been observed in crystal quality. Quality is judged by observation of a crystal in polarized and laser light. At the beginning all the crystals contained defects of a very gross nature. The most recent runs have contained fewer defects but these do permit some degradation in optical quality. However even the smaller defects have caused the crystals to crack as they approached final diameter or shortly thereafter. It appears that a satisfactory furnace design is being approached since a recent growth run contained no defects. This growth run ended prematurely when the power supply failed and the crystal cracked while quenching to room temperature. Reliability of the growth equipment remains high, however, and

should not affect future results significantly.

3.1 Early Run Examples

Table I summarizes the growth runs completed since initiation of the growth program. The first three growth attempts were designed to evaluate the control system on the new power supply. The smaller 4 inch diameter crucible was utilized with a standard growth station design in order to make a comparison with results of growth runs made prior to initiation of the program. Some problems were experienced with melt contamination which originated from flaking of the quill used for holding the seed rod. This was caused by the higher temperature in the vicinity of the quill. Once the problem was identified the diameter control system functioned normally although poor control resulted from the effects of the contamination.

For subsequent growth runs the larger 4.5 inch diameter crucible was put into service. The growth run number is out of sequence with other runs in the table because the crucible was already charged from an earlier growth run attempted with a smaller power supply. In this earlier attempt the generator had enough power to fully melt the charge but there was not enough residual power to initiate growth when the seed was dipped. This clearly indicated that the larger 50 kW power supply was required to conduct growth with the 4.5 inch diameter crucible. A small section of crystal was grown, but

Table I

Summary of Crystal Growth Runs

Growth Run	Crucible Size (in.)	Crystal Dia. (in.)	Pull Rate (in./hr.)	Rot. Rate (RPM)	Comments
N2141	4 x 4	1.40	0.020	15	No crystal. Run terminated due to melt contamination.
N2154	4 x 4	1.40	0.020	15	No crystal. Run terminated due to melt contamination.
N2162	4 x 4	1.40	0.020	15	No crystal. Run terminated due melt contamination.
N2014	4.5 x 4.5	1.75	0.020	15	Poor diameter control. Atmosphere control off due to bell jar leak.
N2227	4.5 x 4.5	1.75	0.020	15	Three growth attempts with cracking. Final attempt with extended length at small diameter.
N2301	4.5 x 4.5	1.75	0.020	15	Three growth attempts with cracking. Small blossoms in small diameter section.
N2329	4.5 x 4.5	1.75	0.020	15	Two growth attempts. Crystal cracked when power failed. No. blossom.

the diameter control was poor (Figure 3) due to problems with growth atmosphere control. It was later learned that a crack had developed in the fused silica bell jar used to enclose the growth furnace.

The final three growth runs were performed with the water-cooled bell jar system. This approach was found to work very well since the excessive heat evolved from the growth furnace was effectively conducted away by the water-cooled enclosure. Without this system it would have been impossible to work in the vicinity of the growth furnace and the heat would have had a deleterious effect on the electronic control system.

3.2 Growth at Seed Diameter

Experience in the production growth of Nd:YAG indicates that the best results are obtained if the crystal is allowed to grow with a very steep solid/liquid interface projecting down into the melt. While this highly convex shape results in a core formation from facets developed at the tip of the growth interface, most of the strain is confined to a 3-4 mm diameter core region. High quality laser rods can then be extracted from the outer portion of the crystal cross-section and in between the radial strain lines.

An unfortunate consequence is that disturbance of the crystal diameter normally results in a blossom (local high

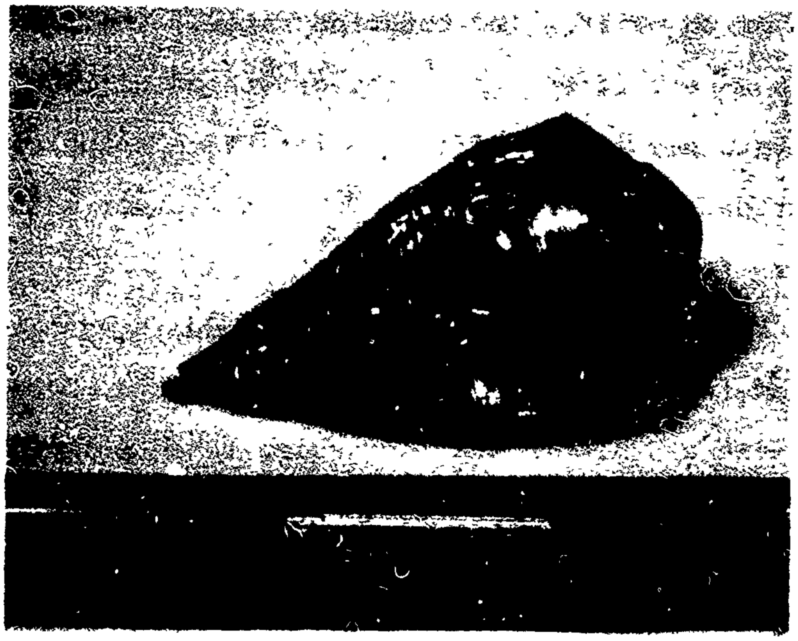

Figure 3   Crystal Started with Poor Diameter Control

strain) fanning out from the central core. This situation also exists if the growth interface is not convex enough since the faceted central region then has a tendecy to trap liquid or secondary phases which result in defects.

Methods of insuring that the crystal maintains a highly convex profile are either to provide large temperature gradients or to utilize low rotation rates. The latter method alone is not very effective in the growth of Nd:YAG, since the rotation rate has little effect on interface shape except at high rotation rates (~100 RPM). Thus the former method is resorted to for production growth.

For the situation which exists during growth of the larger diameter crystals care must be exercised, because the crucible size and growth station design tend to increase the temperature gradients. Efforts to increase the existing gradients can lead to cracking when the yield strength of the crystal is exceeded.

Most of the initial work during this program favored crystals which contain blossoms arising from a shallow interface shape. In many cases the resulting strain was so gross that extensive cracking of the crystals resulted. Blossom formation occurred at diameters of 0.5-1.0 inch. An alternate method of lengthening the growth interface was attempted and early results indicate that some improvement in growth occurred.

In this instance initial growth was conducted at somewhat larger than seed diameter for an extended length and the crystal diameter was then increased slowly to its final value (Figure 4). It was felt that the additional heat sink capacity of the extended length of small diameter crystal would provide a steeper growth interface and therefore overcome the blossom formation in the 0.5-1.0 inch diameter range. Radiative losses could then maintain the steep interface as the crystal diameter was increased. Whether an improvement in growth results from this approach or small changes in growth station design is not clear yet.

3.3 Measured Melt Gradients

It is felt that the most important parameter requiring control during the growth of Nd:YAG is the radial melt temperature gradient. Because of the crystal's high melting point (1950°C) it is difficult to measure this directly by accurate methods. One approach which has been utilized satisfactorily is to scan the melt surface with the optical pryrometer used for diameter control. This method has been found to be repeatable and has proved to be useful for qualifying the growth results of various growth station designs.

Figure 5 illustrates the results of two such scans for different growth station designs. Although similar in some respects, these charts have one characteristic which may be related to blossom formation at small crystal diameters.

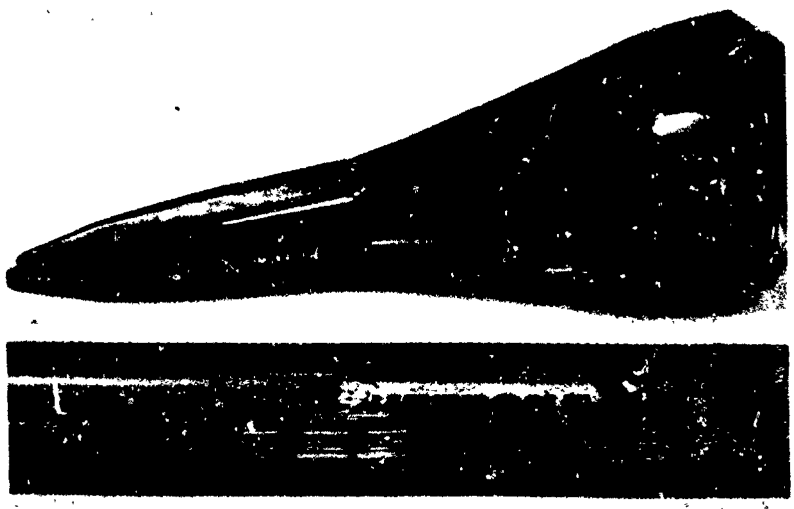

Figure 4 Growth From Slowly Increased Seed Diameter for 2 Inch Length

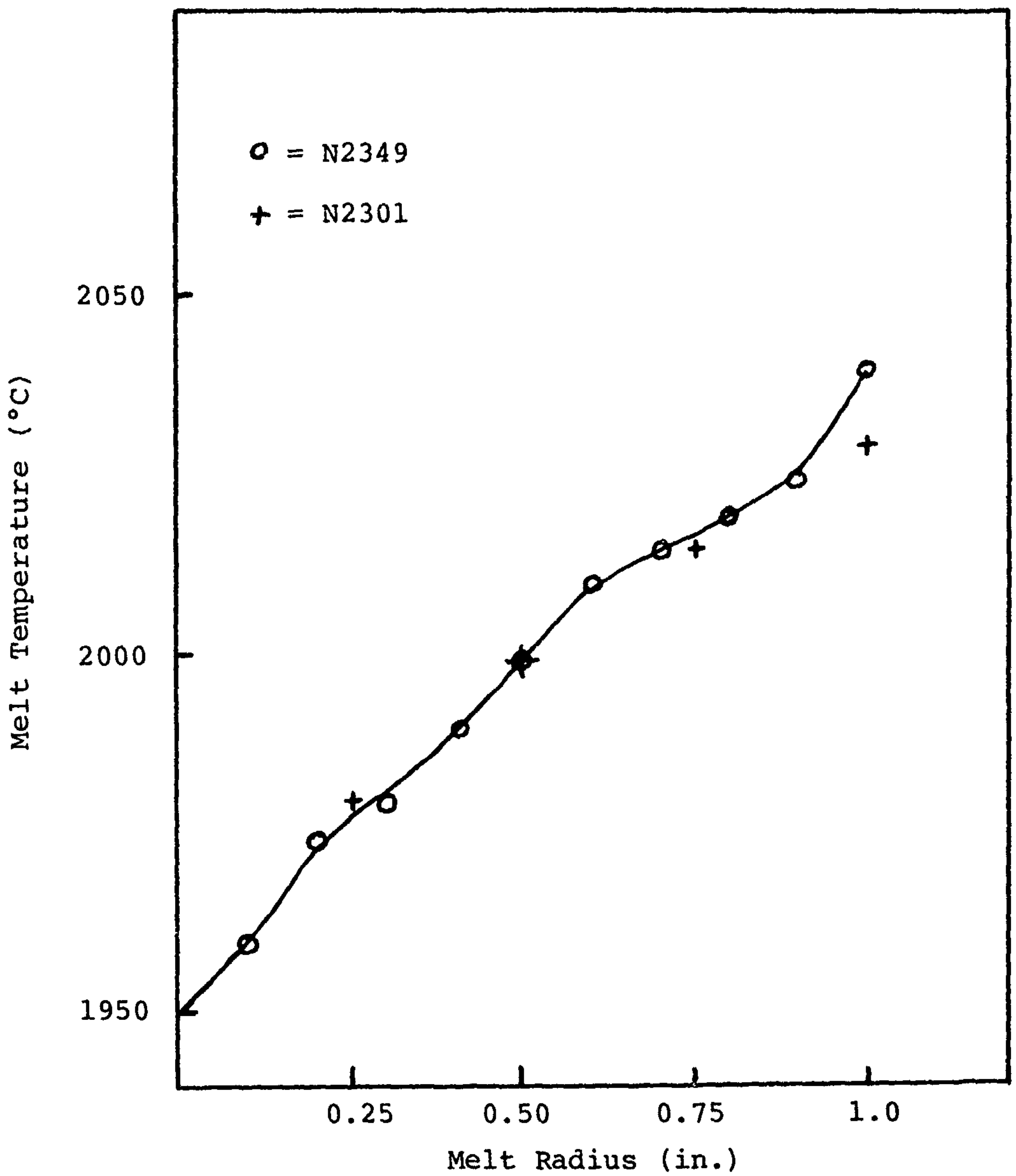

Figure 5 Nd:YAG Melt Temperature as a Function of Melt Radius

It can be seen that the radial melt temperature gradient is higher at smaller melt diameter and then reduces as the distance from the melt center increases. This means that constitutional supercooling can occur at small crystal diameter if the rate of the diameter increase exceeds the ability of the diameter control system to maintain the crystal on its program. Ideally the radial gradient should have a low slope more typical of that observed at the larger radii in Figure 5.

3.4 Recent Runs

Significant progress was made in the final two growth runs outlined in Table I. The reason for this is believed to be a combination of improved growth station design and growth of an extended length of crystal at small diameter prior to diameter expansion.

Although practically all of the crystals grown during this program contained blossoms at small diameter, an improvement in crystal quality resulted as soon as the crystals were grown for an extended time period at small diameter. The gross strain and severity of cracking appeared to be reduced following the appearance of a blossom. Use of the water-cooled growth station enclosure was initiated with growth run 2301 and a further improvement in quality was observed. However, this may have resulted from a growth station design change which was made concurrently.

The final growth run (No. 2329) made during the reporting period was of excellent quality. Unfortunately the power supply failed shortly after the crystal attained final diameter. Although the crystal cracked while quenching to room temperature, it was possible to examine it closely between crossed polarizers. The lack of internal defects was attributed primarily to the smooth diameter control and secondarily to the station design change.

3.5 Problem Analysis

The major difficulty which has prevented growth of larger diameter crystals of high quality has been the internal blossom generation at small crystal diameter. This has led to an inordinate amount of strain in most cases and finally the crystal cracks. However, even in the cases where the strain from blossom generation is comparable to that encountered in production growth of smaller diameter crystals, cracking has occurred. It would appear, therefore, that the larger diameter crystals are unable to withstand the greater differential thermal stress between the cold surface and hot center of the crystal. A simple remedy for this appears to be a top heater.

The most recent growth results indicate that defect-free crystals of larger diameter should be obtainable. While improving the growth station design, attention will have to be given to the area above the crucible into which

the crystal is pulled during growth. Better insulation of this chamber should reduce the thermal loss and thus the differential thermal stress and tendency toward cracking. However, this is expected simultaneously to affect the radial and axial gradients so that blossom generation is prevented and thermal shock is alleviated.

4.0 Conclusions

Results of the first six months of crystal growth indicate that growth of crystals approaching two inches in diameter should be possible. The difficulty in accomplishing this objective is greater than anticipated, however. It is expected that as familiarity with these large growth systems is improved, more consistent results will be obtained in the growth program. Initial results indicate that melt characteristics are similar to those which exist in production growth of smaller diameter crystals. Thus a fine tuning of our current techniques and station designs is required.

One significant drawback is a limitation in equipment devoted to crystal growth coupled with the long time span for completion of a growth run. This seriously limits the rate at which changes can be made to improve portions of the growth process. In addition the rate at which results of such changes can be interpreted to improve portions of the growth process is affected.

While the objectives of growing a good quality crystal approaching two inches in diameter was nc alized during the first six months of the growth program, considerable progress was made toward attaining this goal. The crystalline quality was steadily improved to the point where a gross degradation in quality did not result after generation of a crystalline defect. This indicates that current station designs are close to that required for achieving consistent results at large diameter.

5.0 Program for Next Period

Further refinement of growth station design and growth technique will be accomplished during the next six month period. It is hoped that growth results will be satisfactory enough that station design can be standardized to the point where refinement of growth procedures can begin.

Fabrication of laser rods from good quality crystals will be attempted to evaluate the quality of such crystals in comparison to production results. It is anticipated that the first delivery of rods from a crystal meeting the size requirements of this program will be made. If this can be accomplished during the next reporting period then verification growth runs will be attempted to qualify the growth process.

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